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Breakthrough pressure is the external pressure required to force a composite – solid, liquid, vapor – interface to transition irreversibly to the fully wetted – solid liquid – interface. Based on theoretical models, this pressure can be calculated using material properties and an understanding of the surface geometry. The inicorporation of low surface energy materials can drastically increase the breakthrough pressure of a material. Cotton fabric was treated in a variety of different tecnoflon / Fluorodecyl$_8$T$_8$ solutions. These textiles were then investigated for geometrical properties by SEM and breakthrough pressure using a unique experimental apparatus. Resulting data was compared with theoretical model predictions.

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Introduction

There has been a vast amount of research recently directed at obtaining superhydrophobic and superoleophobic textured surfaces. Geometrical parameters based on these textures have been developed to model predicted contact angles with liquids of varying surfaces tensions. One way of determining the robustness of the superhydrophobic state is to study the amount of external pressure required to force the wetting of the material. This sudden and irreversible transition from a Cassie-Baxter state to the Wenzel state happens at a critical point known as the breakthrough pressure[1]. Fabrics, due to the weave of the individual fibers and bundles, have a fairly regular reentrant cylindrical structure which can readily be applied to these models. For such a structure, the resulting breakthrough pressure can be given by the following:

$$P_{bt} = \frac{D^2 + 2DR\sin^2\theta}{Rl_{cap}(1-\cos\theta)(\sin\theta)}$$

Where: 2D is the spacing between features, R is the radius of those features, and θ is the apparent contact angle of the probing liquid on a smooth surface of the same material. Using this model, breakthrough pressure could be increased in several ways. Altering of the geometrical spacing of the sample weave has been investigated by applying biaxial strain while in contact with various probing solutions[2]. By applying a coating of high contact angle material, the breakthrough pressure of the fabric samples should be increased. In this instance, fluorodecyl$_8$T$_8$ POSS (hereafter referred to as FD8T8) and a commercially available fluoropolymer – Tecnoflon – were used as the low surface energy modifiers.

Experimental

Materials: Fabric samples were all from a single 100% cotton shirt purchased at a commercial retailer. Asakilin AK-225 was purchased from AGC Chemicals Americas. Tecnoflon BR9151 was obtained from Solvay-Solexis. Silpak Silicone rubber RTV was obtained from Silpak Inc. Fluorodecyl$_8$T$_8$ POSS was synthesized internally. The synthesis and characterization of this material can be found in Mabry et al[4].

Figure 1. Experimental setup to determine fabric breakthrough pressure.

Sample Preparation: Four testing solutions were prepared for the dip coating procedure. AK-225 was used as a control, since the solvent could potentially remove some retail coating on the fibers. Tecnoflon and FD8T8 were each dissolved in a separate solution at 1% weight. The final test solution was 1% total solids by weight of a 50:50 ratio of Tecnoflon to FD8T8. Approximately 2 inch diameter sections of the 100% cotton fabric was dipped into each of the solutions and agitated gently. Samples were allowed to remain in the coating solution for 5 minutes. After, samples were dried in an oven at 60^{0}C for 30 minutes. Two samples of fabric were prepared from each testing solution.

Test Procedures: One sample of each testing solution was investigated using SEM. Samples were made as conductive as possible in an effort to obtain the highest resolution SEM images possible. Carbon tape, aluminum tabs, and gold sputtering were all employed on each sample. Initial investigations were used to determine geometrical parameters R and D for use in the breakthrough pressure approximations. Studies of the coated samples were used to confirm that the changes in surface geometry was negligible as well as determine if the applied coating was visible at high magnification.

For hydrostatic breakthrough pressure testing, the samples were placed into two silicone rubber rings, which were then sealed together, leaving an exposed circular surface of the fabric sample. These sample holders were then fixed into a cylindrical glass tube, with around several inches of tubing above the sample ring (see figure 1). Water was slowly added to the top of the glass tube until water began to flow through the exposed fabric surface. The height of the water was measured at this point. Each sample was tested, dried and then tested again.

Results and Discussion

Investigation by SEM showed the geometrical parameters of interest readily. Two separate values were recorded and used to determine which was more appropriate. The spacing between individual fibers, or the bulk spacing between the bundles. These values were consistent for all samples, treatment with the dip coating solutions did not appreciably change the geometry. For the fiber bundles, 2d is 110 μm, and 2R is 350μm (see figure 2). In the case of the single fibers, 2D is 30 μm and 2R is 2μm. Using these values and a contact angle of 125^0 for a smooth surface of either FD8T8 or Tecnoflon, approximated breakthrough pressures were obtained. For the case of the individual fibers, the predicted pressure is ≈600 Pa, or 2.5 inches of water. If considering the fiber bundles, the breakthrough pressure approximation increases to ≈ 1000 Pa or 4 inches of water.

Figure 2. SEM Image of untreated cotton fabric surface. Geometrical parameters of interest are labeled.

Breakthrough pressures were successfully obtained for nearly all samples tested. AK-225 treated fabric was less effective than completely untreated cotton. As is apparent in figure 4, the higher of the two estimations for breakthrough pressure appears to be more accurate. This means, the breakthrough pressure in this case is driven by the larger scale features.

Interestingly, the samples appeared to deform slightly between their two breakthrough pressure tests. This did not have a readily apparent effect on the outcome of the tests. To further investigate the possibility, several samples were tested with a wire mesh support beneath the fabric sample. In all cases the obtained values from the pressure test were unchanged.

Figure 3. SEM Images of individual fibers in the fabric mat: untreated (left) and tecnoflon/fluoroPOSS dip coated (right).

SEM images showed very little difference between untreated and dip coated samples. At high magnification it was apparent that the treated samples had some order of additional surface roughness, as can be seen in figure 3.

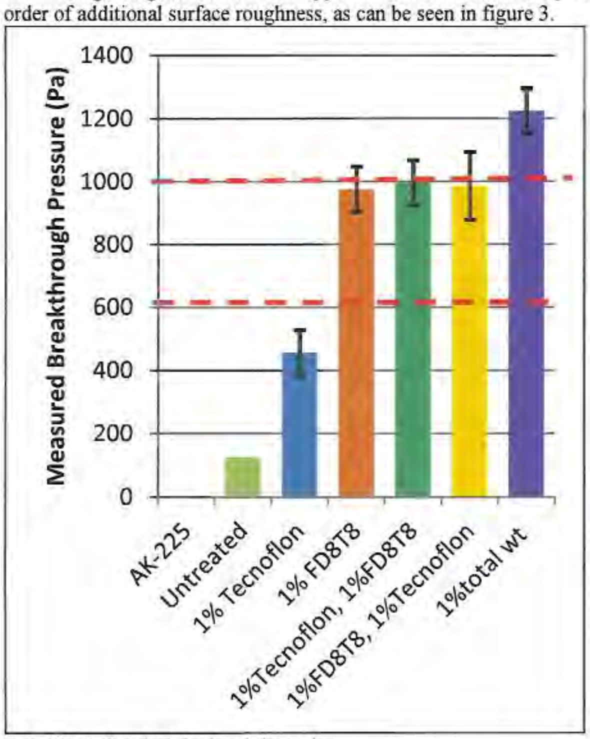

Figure 4. Chart showing the breakthrough pressure

Conclusions: Model predictions for breakthrough pressure of materials can be used under the right circumstances to effective predict the pressure at which wetting will occur. Coating cotton fabric with low surface energy materials can significantly increase the breakthrough pressure of liquids. This does not noticeably change the geometrical parameters of the fibers, so the effectiveness of the treatment derives from the materials properties of the coating.

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